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Optimization of Biodiesel Production from Tallow Oil

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ABSTRACT
In this work, the production of biodiesel from beef tallow has been investigated. The biodiesel was produced via transesterification of beef tallow in the presence of NaOH catalyst. The feed stock (Tallow) was initially pretreated and then characterized. The characterization was followed by esterification of the tallow oil in order to reduce the FFA level prior to transesterification. During the transesterification, the effect of key parameters such as temperature, reaction time, catalyst concentration and methanol-oil ratio were investigated. These process parameters were analyzed using the Response Surface Methodology (RSM) and analysis of variance (ANOVA). The combined effects of the process parameters and their level of significance were investigated using the Central Composite Design (CCD) of the RSM. The results obtained are in good agreement with other published data of biodiesel production from animal fats as well as various international standards for biodiesel fuel. A second order model was obtained to predict the yield as a function of the process parameters. An optimum yield of 93.86% was achieved at optimum conditions of temperature of 65 C; reaction time of 60 minutes; methanol-oil ratio of 6:1 and catalyst concentration of 0.5wt%. The physical properties of the biodiesel were determined as: density 883.38kg/m^3, viscosity 4.59mm^2/s, flash point 150^{o}C, cloud point 14^{o}C, pour point 8^{o}C, acid value 0.421mg KOH/g.

KEY WORDS: *Biodiesel, transesterification, triglyceride, tallow, methanol, response surface methodology (RSM), central composite design (CCD), analysis of variance (ANOVA).*

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I. INTRODUCTION

Considering the fact that the supply of fossil fuels will decrease in future as the energy demand will continue to grow rapidly, the search for alternative renewable fuels has gained fundamental importance. Also, the environmental concerns regarding greenhouse gas emissions and the commitment of the international community to significantly reduce emissions, tagged the need to find more sustainable alternatives to fossil fuels.

Biodiesel which is fatty acid esters obtained by transesterification of vegetable oils or animal fats is one of these alternatives. Animal fats are attractive feedstock for biodiesel because their cost is substantially lower than the cost of vegetable oils. This is largely due to the fact that there is abundance of animal fat in the world (about 17,500tonnes produced per year in Nigeria, for example) and much of the fats are considered inedible by humans. More so, there is very few competition for the use of animal fats hence its market is limited.

Biofuels are alternatives to the sole dependence on oil. It presents another source to rely on if there is a break in the supply of oil and also it relieves the stress on oil consumption. Increase in biofuel production would mean an increase in plant and animal production since more feedstock would be required, which would also increase jobs. This is very important, especially for developing countries where the rate of unemployment is high.

This research studies biodiesel production from tallow and optimization of its variables. Biodiesel is one such biofuel that is produced using the transesterification process. Biodiesel is a diesel replacement fuel for use in compression ignition engines. It is manufactured from plants oils (soybean oil, cotton seed oil, canola oil), recycles coking greases or oils (e.g. yellow grease), or animal fats (beef tallow, pork lard). The biodiesel manufacturing process converts oils and fats into long-chain mono alkyl esters, or biodiesel.

This research paper focuses on the production of biodiesel from tallow oil through transesterification using sodium hydroxide as catalyst, and the optimization of the process parameters using a central composite design (CCD) of response surface methodology (RSM).

II. MATERIALS AND METHODS

2.1 Material acquisition

The tallow used for the experiment was collected from an abattoir located at Obinze cattle merchant settlement in Owerri West L.G.A. of Imo State. The raw tallow samples were collected from the slaughter arena and put into a well labeled nylon sack and taken to New Concept Analytical Laboratory Obinze. At the laboratory the pretreatment of the Tallow was done prior to transesterification.

2.2 Preparation of Tallow

The weighed tallow (2500g) was heated to a temperature of about 120ºC for 30 minutes, in order to melt the fat, and to remove moisture together with other impurities present. The well heated/melted fat (tallow) is allowed to cool to a temperature of 50ºC and then filtered through a sieve cloth in order to recover the fat which is used for the experiment analysis/tests.

2.3 Acid Pretreatment (Acid Catalyzed Esterification)

The tallow was weighed and then heated at 60ºC for about 10mins and mixed with (60% w/w oil) methanol. To the mixture, was added 1.2% w/w of concentrated H_2SO_4.

The resulting mixture was then stirred on magnetic strip plate for 1hr at 60ºC after which it was allowed to settle for the methanol-water phase at the top.

2.4 Design of Experiment

Montgomery (2003) mentioned that by designed experiment, engineers can determine which subset of the process variables has the greatest influence on process performance. The results of such an experiment can lead to:

1. Improved process yield
2. Reduced variability in the process and closer conformance to nominal or target requirement.
3. Reduced design and development time.
4. Reduced cost of operation.

Several approaches can be considered in running an experiment. The central composite design (CCD) of the Response Surface methodology (RSM) was used for this study. The CCD is an experimental design used for building a second order model for responses without the need to use a complete three level factional experiment and provides information about interactions among experimental variables within the range of studies, leading to better knowledge of the process (Box & Wilson, 1951).

Based on the CCD experiments were carried out to optimize the parameters. The independent variables in the range are the methanol-oil ratio, the catalyst concentration (%wt of oil/fat), reaction temperature (ºC) and time (mins) to achieve high value of biodiesel yield (%), which is set as the measurable response factor.

Exactly 50g of Tallow oil was measured out and poured into the reactor. The reaction parameters were modified for each run from the design matrix. The design matrix is as shown in Table 2.1 below.

Table 2.1: Factor notation and amount

Notation	Factors	Low	High
A	Temperature (ºC)	45	85
B	Time (min)	40	80
C	Catalyst concentration (wt%)	0.1	0.9
D	Methanol-oil ratio	4:1	8:1

Table 2.2: Factors and their levels for the central composite design for optimizing biodiesel

Variable	Coding	Unit	Level				
			- 2	-1	0	1	2
Temp	A	DegC	45	55	65	75	85
Time	B	Min	40	50	60	70	80
Cat-Amt	C	Wt%	0.1	0.3	0.5	0.7	0.9
Methanol-Oil ratio	D	-	4:1	5:1	6:1	7:1	8:1

2.5 Alkali-Based Transesterification of Tallow (Biodiesel Production)

The reactor was filled with the desired amount of pretreated tallow and placed on the magnetic stirrer with its associated equipment. The pretreated tallow was then agitated at 1200rpm and heated to a predetermined temperature (45-85ºC) depending on the experimental run. The catalyst, NaOH was mixed with methanol (amount by mass based on the experimental design run) in a conical flask, and the resulting solution (sodium methoxide) was added to the tallow oil in the reactor. The reaction was turned as soon as the sodium

methoxide was added to the reactor and it continued for the required time. At the end of the stipulated time, two layers were formed: the upper layer was made up of methyl esters, some methanol and traces of NaOH while the lower layer was made of glycerol, most of the catalyst and methanol. The mixture was allowed to stand undisturbed for 10 hours before the ester phase was separated.
Several molar ratios of methanol to oil were used for the experimental runs (ranging from 4:1 to 8:1), and within the stipulated time limits of 40-80 minutes.
This experiment was carried out in 30 experimental runs at atmospheric pressure and stirring rate of 1200 rpm, with the factors varied during the experiment in accordance with the outcome of the CCD, within the following ranges.
Temperature: 45 to 85^oC
Catalyst Concentration: 0.1 to 0.9 wt%
Methanol-Oil Ratio: 4:1 to 8:1
Time: 40 to 80 minutes

2.6 Separation of Biodiesel from other Products

The mixture was transferred to a separation funnel, allowing glycerol to separate by gravity for 10 hours, after removing the glycerol layer (the lower layer), the methyl ester layer was then cleaned thoroughly by washing with warm (50^oC) de-ionized water to remove methanol, the catalyst and glycerol residuals. The methyl ester (biodiesel) was then dried in the oven at 110^oC for two hours. The quantity of dried biodiesel obtained for each experimental run was noted and the percentage yield calculated.

III. RESULTS AND DISCUSSION

3.1 Results presentation

The results of the experiments carried out at the different process parameters on the production of biodiesel from tallow oil is tabularized below:

Table 3.1:Central composite design, experimental and predicted values of biodiesel yield

Std	Run	Temp (DegC)	Time (min)	Cat-amt (wt)	Ratio	Exp (%)	Predicted (%)
1	7	55	50	0.3	5	83.8	84.06
2	22	75	50	0.3	5	84.4	83.32
3	16	55	70	0.3	5	83.0	82.45
4	9	75	70	0.3	5	82.05	81.99
5	25	55	50	0.7	5	80.5	79.47
6	5	75	50	0.7	5	86.6	87.24
7	30	55	70	0.7	5	78.0	77.67
8	29	75	70	0.7	5	84.3	85.71
9	1	55	50	0.3	7	91.5	88.81
10	12	75	50	0.3	7	82.0	82.84
11	24	55	70	0.3	7	85.4	84.85
12	13	75	70	0.3	7	79.0	78.75
13	21	55	50	0.7	7	89.8	89.95
14	19	75	50	0.7	7	92.8	92.07
15	14	55	70	0.7	7	86.0	85.80
16	18	75	70	0.7	7	88.87	88.20
17	20	45	60	0.5	6	88.1	89.72
18	27	85	60	0.5	6	91.82	91.38
19	2	65	40	0.5	6	86.2	87.38
20	4	65	80	0.5	6	81.9	81.91
21	10	65	60	0.1	6	78.5	79.91
22	26	65	60	0.9	6	85.0	84.78
23	11	65	60	0.5	4	77.5	77.03
24	17	65	60	0.5	8	82.6	84.26
25	6	65	60	0.5	6	92.84	93.39
26	15	65	60	0.5	6	93.86	93.39
27	23	65	60	0.5	6	93.86	93.39
28	8	65	60	0.5	6	93.86	93.39
29	28	65	60	0.5	6	93.84	93.39
30	3	65	60	0.5	6	92.04	93.39

Table 3.2: Analysis of Variance (ANOVA) for Response Surface Quadratic Model
Analysis of variance table (Partial sum of squares)

Source	Sum of squares	DF	Mean Square	F Value	Prob> F	Remarks
Model	793.77	14	56.70	32.00	<0.0001	Significant
A-Temperature	4.13	1	4.13	2.33	0.1475	

B – Time	45.05	1	45.05	25.42	0.0001	
C – Cat Amt	35.58	1	35.58	20.08	0.0004	
D – MeOH:Oil	78.55	1	78.55	44.34	<0.0001	
A^2	13.79	1	13.79	7.79	0.0137	
B^2	131.15	1	131.15	74.02	<0.0001	
C^2	209.19	1	209.19	118.07	<0.0001	
D^2	278.53	1	278.53	157.20	<0.0001	
AB	0.078	1	0.078	0.044	0.8362	
AC	72.34	1	72.34	40.83	<0.0001	
AD	31.87	1	31.87	17.98	0.0007	
BC	0.038	1	0.038	0.021	0.8855	
BD	5.50	1	5.50	3.10	0.0985	
CD	32.83	1	32.83	18.53	0.0006	
Residual	26.58	15	1.77			
Lack of fit	23.72	10	2.37	4.15	0.0648	Not significant
Pure Error	2.86	5	0.57			
Cor Total	820.35	29				

Table 3.3: Statistical Goodness of fit for the Quadratic Model

StdDev	1.33	R-Squared	0.9676
Mean	86.32	Adj R-Squared	0.9374
C. V.	1.54	Pre R-Squared	0.8284
PRESS	140.74	Adeq Precision	17.387

Table 3.4: Regression Model for the Experimental Data

Factor	Coefficient estimate	DF	Standard error	95% CI Low	95% CI High
Intercept	93.39	1	0.54	92.23	94.55
A-Temperature	0.42	1	0.27	-0.16	0.99
B-Time	-1.37	1	0.27	-1.95	-0.79
C-Cat Amt	1.22	1	0.27	0.64	1.80
D-MeOH:Oil	1.81	1	0.27	1.23	2.39
A^2	-0.71	1	0.25	-1.25	-0.17
B^2	-2.19	1	0.25	-2.73	-1.64
C^2	-3.19	1	0.25	-3.30	-2.33
D^2	-3.19	1	0.25	-3.73	-2.64
AB	0.070	1	0.33	-0.64	0.78
AC	2.13	1	0.33	1.42	2.84
AD	-1.41	1	0.33	-2.12	-0.70
BC	-0.049	1	0.33	-0.76	0.66
BD	-0.59	1	0.33	-1.30	0.12
CD	1.43	1	0.33	0.72	2.14

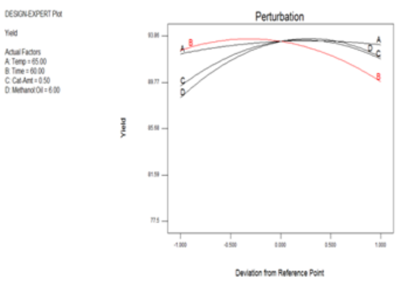

Fig3.1:Perturbation plot of the process parameters

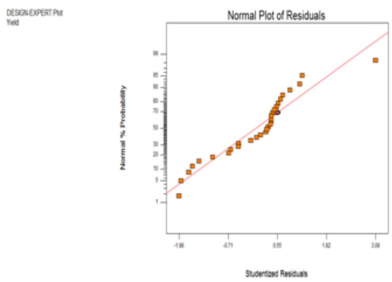

Fig 3.2:Normal probability plot

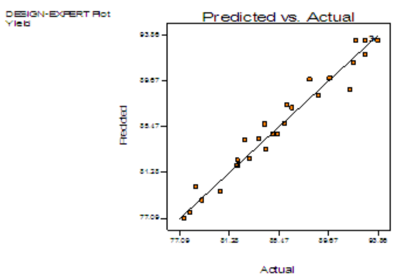

Fig 3.3:Plot of predicted and actual biodiesel yield

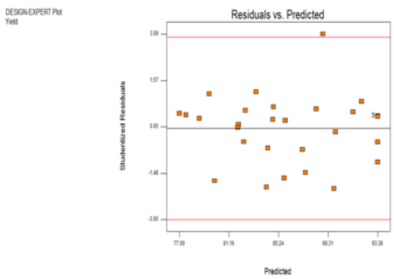

Fig 3.4: Plot of residual and predicted biodiesel yield

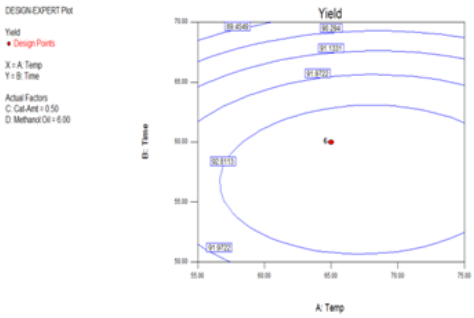

Fig 3.5: Contour plot of time versus temperature

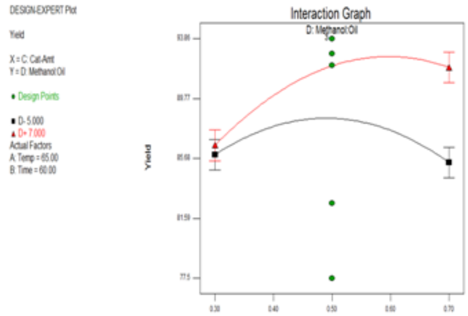

Fig 3.6: Plot of interaction of catalyst amount and methanol-oil ratio

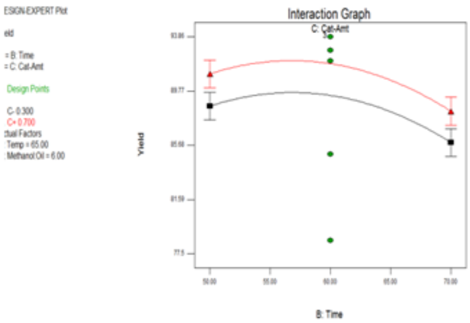

Fig 3.7: Plot of interaction of time and catalyst Amount

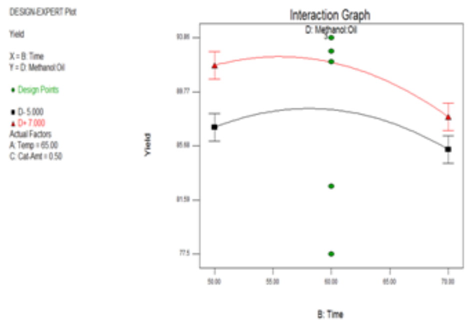

Fig 3.8: Plot of interaction of time and methanol-oil ratio

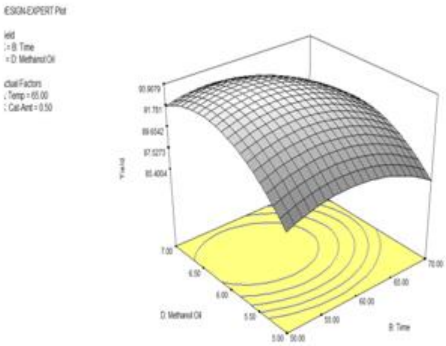

Fig 3.9: Response surface plot of time and methanol-oil ratio

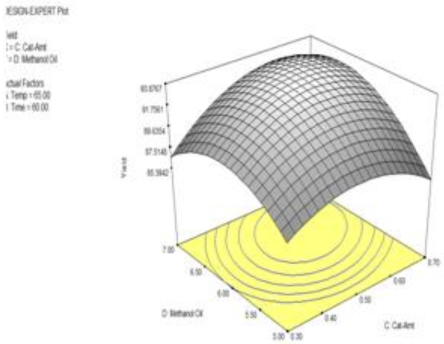

Fig 3.10: Response surface plot of catalyst amount and methanol-oil ratio

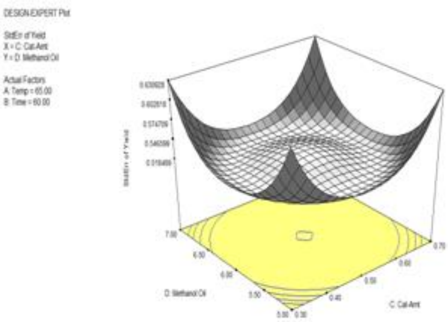

Fig 3.11: Standard Errorplot of catalyst amount andmethanol-oil ratio

Table 3.5: Physical Properties of the Produced Biodiesel

Properties	Produced Biodiesel	Standards for Biodiesel EN14214
Density at 15 C kg/m^3	883.38	860-900
Viscosity at 40ºC mm^2/s	4.59	3.5-5.0
Flash point ºC	150	101 Min
Cloud point ºC	14	-
Pour point ºC	8	-
Acid Value mg KOH/g	0.421	0.5 Max

Table 3.6: Optimal Results Validation

Variable	Model-derived optimum value (experimental)	Experimentally-derived optimum value (validation)
Temperature , A (deg C)	65	65
Time, B (mins)	60	60
Catalyst amt, C (%wt)	0.5	0.5
MeOH-Oil ratio, D	6:1	6:1
Yield, Y (%)	93.86	82.80

3.2 Discussion of Results

30 experiments were performed to get the experimental values of the biodiesel yield of Tallow. Experimental and predicted values for the biodiesel yield responses at the design points are given in table 3.1.

3.2.1 Statistical Analysis

The design expert 6.06 software was used for the regression and graphical analysis of the data. The maximum values of biodiesel yield were taken as the response of the design experiment. The experimental data obtained by the above procedure was analyzed by the response surface regression using the following second-order polynomial equation.

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum_{i>j}^{k} \sum_{j}^{k} \beta_i x_i xj \qquad (3.1)$$

where y is the response (Biodiesel yield), x_i and x_j are the uncoded independent variables, i and j are the linear and quadratic coefficients respectively, β_0 is the regression coefficients, k is the number of factors studied and optimized in the experiment. Statistical analysis of the model equation and of the analysis of variance were carried out.

From Table 3.3, the "Pred R-squared" of 0.8284 is in reasonable agreement with the "Adj R-Squared" of 0.9374. "Adeq Precision" measures the signal to noise ratio. This model can be used to navigate the design space.

3.2.2 Analysis of variance (ANOVA)

Analysis of variance was applied on the data to estimate the effect of main and interactive effects of the factor combinations on the yield. From Table 3.2, it was clear that apart from temperature, all the other three factors under examination as well as the model were statistically significant $P < 0.001$ generally, P-values lower than 0.05 indicate that the overall model is significant due to large number of individual significant terms which is desirable in indicating that they have effect on the response and was sufficient to response and the independent variables.

3.2.3 Interaction Effects

The effect of independent variables on the yields were also accompanied by the effects of their interactions. These are the effects due to multiples of one factor and another or the same factor. These interaction factors must be considered as the individual plots does not give information regarding the significant interaction involved. These effect resulting from the multiples of either the same factors or two factors are known as compounding or interaction effects. From the ANOVA (Table 3.2), it was visible that the interaction of the reaction temperature and catalyst concentration (AC), temperature and methanol: oil ratio (AD) and catalyst concentration and methanol: oil ratio (CD) were significant for the first order polynomial of the independent factor combinations while the rest of the first order terms were not significant implying that the terms have no effect on the response (Yield); while the interactions between temperature and reaction time (AB) was insignificant. The interaction effects between reaction time and catalyst amount (BC) as well as that between reaction time and methanol to oil molar ratio (BD) had no significant effect on the yields.

In addition, the second order polynomial terms were all statistically significant at different levels of significance. This signified that the so called insignificant interactions (AB, BC and BD) in the first order are actually significant as the sample population increases. However, being significant does not define positive or negative

effect on yield, rather it means that when such a term is increased or decreased a corresponding change in the response is expected. From the results, it could be suggested that factors B, C and D have much effect on the yield than A. Figure 3.1 is the perturbation plot of the process parameters. It shows the effect of catalyst concentration, reaction temperature, reaction time and methanol to oil molar ratio. From the figure, factor A (temperature) has little effect on the biodiesel yield. The response decreases with increase in reaction time (factor B). For factor C (catalyst concentration), the yield increases significantly with increase in its concentration and then decreases significantly. This may be due to the fact that addition of excessive catalyst causes more triglyceride to react with the alkali catalyst concentration leading to the formation of soap which decreases the yield. The biodiesel yield is found to increase with increase in methanol/oil ratio, since the transesterification reaction is reversible in nature, so excess alcohol is added to ensure the total conversion of triglycerides.
In order to determine the positive and negative contributions of independent and interactive terms in the model, the response equation is employed to calculate both the coded and un-coded values of the response.

3.2.4 Response Equation (Developing a regression Model)

The conversion between the experimental process variables and yield was evaluated using the CCD modeling technique of design expert version 6.06.
Second order polynomial regression equation was fitted between the response (yield) and the process variables: reaction temperature (A), Time (B), Catalyst amount (C) and Methanol/oil molar ration (D). From Table 3.2, the ANOVA results showed that the quadratic model is suitable to analyze the experimental data (Sahoo& Das, 2009). The predicted model for percentage of biodiesel (Y) in terms of the coded and un-coded factors of the process variables is generated below. Their various confidence level are also shown in Table 3.5.
Final equation in terms of coded factors:

$$\text{Yield} = 93.39 + 0.42A - 1.37B + 1.22C + 1.81D - 0.71A^2 - 2.19B^2 - 2.76C^2 - 3.19D^2 + 0.07AB + 2.13AC - 1.41AD - 0.049BC - 0.59BD + 1.43CD \quad (3.2)$$

Final equation in terms of actual factors:

$$\text{Yield} = -17.3.75323 + 1.23660\text{Temp} + 2.80544\text{Time} - 35.48646\text{Cat Amt} + 49.15854\text{ MeOH:oil} - 7.09167E - 0.03\text{Temp}^2 - 0.021867\text{ Time}^2 - 69.04167\text{ Cat amt}^2 - 3.18667\text{ MeOh:oil}^2 + 7.0000E - 000\text{ Tem x Time} + 1.06312\text{ Temp x Cat Amt} - 0.14113\text{ Temp x MeOH:Oil} - 0.024375\text{ Time x Cat Amt} - 0.058625\text{ Time x MeOH} + 7.16250\text{ Cat Amt x MeOH} \quad (3.3)$$

The significant of the regression coefficients was evaluated based on the P-values. The coefficient term with P-values more than 0.05 are insignificant and are removed from the regression model. The analysis Table 3.2 shows that linear terms of time, catalyst concentration and methanol: oil ratio, quadratic terms of temperature, time, catalyst concentration and methanol: oil ratio and interactive terms of temperature and catalyst, temperature and methanol: oil ratio, catalyst and methanol: oil ratio that is B, C, D, AC, AD, CD, A^2, B^2, C^2, D^2, are significant model terms.
The model reduces to:

$$\text{Yield (Y)} = 93.39 - 1.37B + 1.22C + 1.81D + 0.71A^2 - 2.19B^2 - 2.76C^2 - 3.19D^2 + 2.13AC + 1.41AD + 1.43CD \quad (3.4)$$

The analysis of variance shown in Table 3.2 indicated that the quadratic polynomial was significant and adequate to represent the actual relationship between the yield and the significant model variables as depicted by very small p-value (<0.0001).

3.2.5 Reliability of the Model

In determining the model reliability, the R^2 value also known as determination or regression coefficient is important indicating the model fitness. A high value of the predicted R^2 value is an indication of precision. The "predicted R-Squared" of 0.8284 is in reasonable agreement with the "Adjustable R-Squared" of 0.9374. The R^2 value of 0.9676 shows that only 3.24% of the total variation on the yield could not be explained by the model, the more the value approached unity, the better the model fits the experimental data. Other additional information on the data reliability are the values of CV and the R^2adj (1.54 and 0.9374 respectively).

The probability of finding the optimal point of the actual and the predicted yield is represented by the normal probability plots of residual. The data points are approximately linear indicating normality in the error term. The normal probability plot is shown in Figure 3.2 above.

The graph between the predicted and actual biodiesel yield given in Figure 3.3 shows that the predicted values are quite close to the experimental values, thereby, validating the reliability of the model developed for establishing a correlation between the process variables and the biodiesel yield.

IV. CONCLUSSION

This research studied the production of biodiesel from beef tallow as well as the optimization of its process variables. The biodiesel was produced by the transesterification of beef tallow using sodium hydroxide (NaOH) as the catalyst. The Central Composite Design (CCD) of the Response Surface Methodology (RSM) was used for the optimization of the process parameters which are temperature, reaction time, methanol-oil ratio and catalyst concentration. The RSM showed clearly how the operating conditions of the process was optimized to obtain the maximum yield of biodiesel. The optimized experimental results showed that the optimum conditions for the production of biodiesel from beef tallow are at a temperature of 65^{o}C, reaction time of 60 minutes, catalyst concentration of 0.5%wt and methanol-oil ratio of 6:1. A second order statistical model was obtained for the prediction of the optimum yield of biodiesel as a function of the process parameters. This yield was determined as 93.86%. The results were analyzed based on 95% confidence level of statistical significance. The statistical model developed showed a good agreement between the experimental values and the predicted values thereby demonstrating the usefulness of regression analysis as a good tool for optimization.

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